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THE SCATTER OF MECHANICAL VALUES OF CARBON FIBER COMPOSITES AND ITS CAUSES

S. Roth

Translation of: "Streuung von mechanischen kennwerten von CFK und ihre ursachen", Deutsche Gesellschaft fur Luft- und Raumfahrt, Symposium uber Ermudungsfestigkeit von Flugzeugen und modernen Bauweisen, Darmstadt, West Germany, Sept. 22, 1978, Paper 78-186, pp. 1-20

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16. Abstract

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The scatter of experimental data obtained in an investigation of the parameters of structural components is of vital importance for the design process. The extent of scattering is particularly large for strength parameters which are determined by the resin or the adhesion between fiber and resin. An investigation was therefore conducted regarding the causes of the scatter of the experimental values. Attention is given to an evaluation of the scatter, the scatter and the statistical characteristics of the mechanical parameters of carbon-fiber composites (CFC), and the possibilities which exist to reduce this scatter. It is found that quality control tests with respect to fiber and resin are important for such a reduction.

Introduction /2*

Since the design of structural components must take place with statistically certain characteristic values, not only the average characteristic mechanical values but also the scattering of individual values is of the greatest significance.

In Table 1 the results of long-term measurements taken by Dornier are listed.

TABLE 1. CHARACTERISTIC STATISTIC VALUES OF STRENGTH AND MODULI OF HT FIBER LAMINATES

Material property	No. of samples	Average value $\bar{x}$ [N/mm^2]	Standard deviation σ [N/mm^2]	Variation coefficient $\frac{\sigma}{\bar{x}}$ [%]
Bending strength σ_{bB0°	299	1396	128	9,2
Bending strength σ_{bB90°	394	101	16	15,9
Bending modulus $E_{b\alpha 0^\circ}$	298	108200	12470	11,5
Bending modulus $E_{b\alpha 90^\circ}$	394	7780	430	5,5
Interlaminar shear strength τ_{0°	463	97	16	16,2
Interlaminar shear strength $\tau_{0^\circ/\pm 45^\circ}$	734	74	13	18,2
Fiber content	145	60,8 [V%]	3,7 [V%]	6,0 [V%]

*Numbers in margin indicate pagination of original foreign text.

It turns out that the extent of scattering is considerable, primarily for strengths which are determined by the resin or the adhesion between fiber and resin.

The studies presented below shall serve to clarify the causes of the scattering of these characteristic values. It must be stated that no methodical study was performed, rather the results occurring in a certain time period are classified and compared as much as possible. Through this circumstance, a definite assignment of the influence of parameters was not always possible.

Evaluation of Scattering of Characteristic Values /3

A prerequisite for the evaluation of scattering is knowledge of its distribution. Since most results are normally distributed, it is useful to check distributions of measured values occurring in practice with regard to this. This checking takes place in this study by the cumulant method. For this, we calculate the slope as a measure for symmetry and the curvature as a measure for the steepness of the distribution curve (Fig. 1).

It could be demonstrated that in random sampling from a normally distributed quantity, both the slope as well as the curvature are normally distributed, having an average value of "zero" and a standard deviation which depends only on the number of random samples. This means that the quotient of the slope and standard deviation, or of the curvature and standard deviation, are normally distributed.

Thus we form these quotients and compare them with the factor of normal Gaussian distribution. It turns out here that the quotients are considered to differ from zero only by chance, provided they lie within the 95% range. The limit value u for the statistic certainty of $s = 95\%$ is 1.96 for the normal Gaussian distribution. Therefore, if the quotients are less than 1.96, then the assumption that the values originate from a normally distributed entity cannot be refuted.

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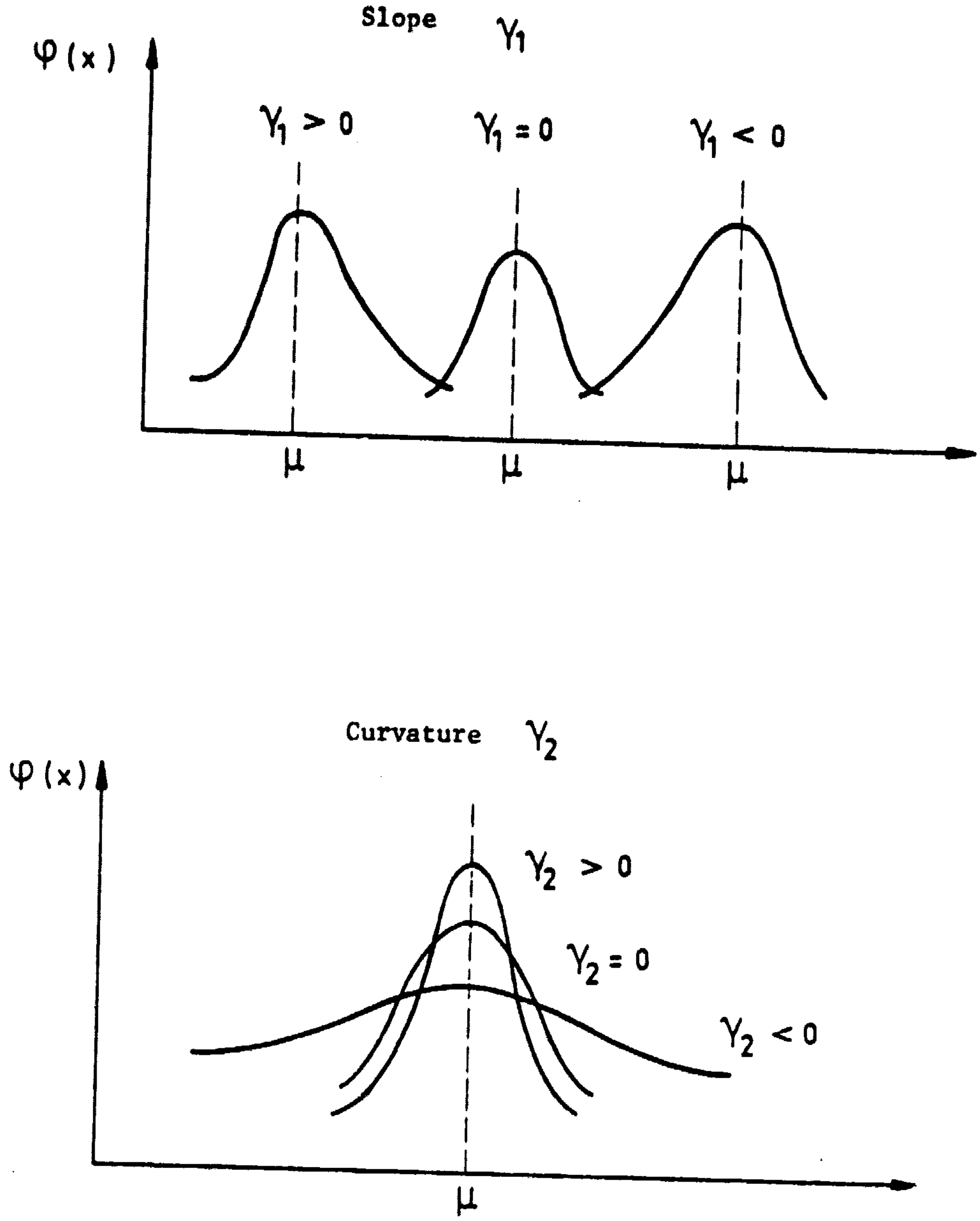

Fig. 1. Slope and curvature of a distribution curve.

Scattering and Statistic Behavior of Characteristic Mechanical Value of CFC /5

In a study of the available results, it was assume that the parameters presented below have an effect on the characteristic values.

-- fiber

-- resin

-- fiber/resin adhesion

-- prepreg preparation

-- handling of the prepreg

-- hardening

The measured results were assigned to the parameters and compared to each other.

A check of the distribution of the measured values from random samples by using the cumulant method showed that a normal distribution cannot be excluded when the random samples studied were taken from one "laminate." The scope of random sampling lay between 6 and 8.

In Tables 2 and 3 statistic characteristic values are presented for strengths determined primarily by the fiber properties and also for strengths determined primarily by the resin properties or by the fiber/resin adhesion. The random samples differ by the fact that they were taken from laminates prepared from prepregs with different fiber charges. By way of explanation, we mention that the following different prepreg charges can exist:

-- equal fiber charge/equal resin charge

-- equal fiber charge/different resin charge

-- different fiber charge/equal resin charge

A prepreg charge is defined so that it must be prepared from equal fiber /6 charge and equal resin charge in a single continuous working process.

Now, as the quotients of slope and curvature of the individual random samples from Tables 2 and 3 show, a normal distribution can be assumed except in two cases. If we consider all measured values, then according to Table 2 this is also true for the cross bending strength but not for the longitudinal bending strength according to Table 1, which already indicates a systematic difference in random samples.

The fact that the values of random samples are normally distributed signifies that conclusions can be drawn from one random sample about all samples, i.e., a check can be made to determine whether the average value of a random sample originates from an assumed entity. This check takes place by means of the so-called t-test. Here, the average value $\bar{x}$ of the random sample is compared with the theoretical value μ of an entity, whereby as variance σ^2 the estimation $\bar{S}x^2$ with f degrees of freedom is available. A number of tests is keyed by the degree of freedom and thus taken into consideration.

We have:

Test size $$t = \frac{\bar{x} - \mu}{Sx} \cdot \sqrt{\mu}$$

The test size t is now compared again with the limit values for the statistic certainty s = 95%.

If $t < t_{95}$, the difference is arbitrary. When $t > t_{99}$, the difference is assured. If we obtain $t_{95} < t < t_{99}$, then the difference is not assured.

For the cross-bending strength (Table 3), the average value of all samples was assumed as the basic entity because the quotients of slope and curvature indicate a normal distribution. /9

In the longitudinal bending strengths, this is not the case; therefore, the average value of random samples from 66 total samples was taken as the basic entity.

As the last column of the tables shows, the deviations of the average values of the random samples from the average value of the basic entity are not always arbitrary, which indicates systematic errors or differences.

If we presume that the parameters "hardening" and "handling of the prepreg" have the same effect for all random samples, then the random samples differ only by the prepreg fiber and partially by the resin batch. Therefore, as causes for systematic differences, only these three parameters come into consideration. Since for three prepreg batches, both the fiber as well as the resin batch were the same, the influence of prepreg preparation on the characteristic

TABLE 2. CHARACTERISTIC STATISTIC VALUES OF LONGITUDINAL BENDING STRENGTH $\sigma_{bBO^{o}}$ OF HT FIBER LAMINATES WITH FIBERS FROM DIFFERENT BATCHES

Mechanical property	Fiber batch	No. of samples	Average value $\bar{x}$ [N/mm^2]	Stand.-deviation s [N/mm^2]	Variation coefficient v [%]	Quotient of slope Schiefe	Quotient of curvature	Deviation $\bar{x}$ from μ
$\sigma_{bBO^{o}}$	C377	18	1358,1	138,1	10,16	+0,386	-0,300	chance
	C380	24	1418,2	110,9	7,82	-1,432	-0,460	chance
	C420	18	1346,2	112,8	8,38	-0,888	+0,397	chance
	2T125B	6	1327,2	57,0	4,30	+1,029	-0,195	highly certain
	2T131CR	66	1390,9[x]	92,1	6,62	+0,637	+0,593	-
	2T131R	24	1446,9	71,3	4,93	-1,065	+0,161	highly certain
	2T137D	113	1380,8	126,8	9,18	-1,955	+2,302	-
	2T146DR	24	1348,6	114,4	8,48	+0,400	-1,149	chance
	2T147E	6	1402,6	45,4	3,24	-0,970	-0,216	chance
	All measured values	299	1395,6	127,6	9,16	+1,503	+5,409	

[x] Average value $\hat{=}$ Average value μ of the entity

Fiber type: 130SC/10 000

Resin type: Code 69

/17

/8

TABLE 3. CHARACTERISTIC STATISTIC VALUES OF CROSS BENDING STRENGTH σ_{bB90° OF HT FIBER LAMINATES WITH FIBERS FROM DIFFERENT BATCHES

Mechanical property	Fiber batch	No. of samples	Average value $\bar{x}$ [N/mm²]	Standard deviation [N/mm²]	Variation coefficient V [%]	Quotient of slope	Quotient of curvature	Deviation $\bar{x}$ from μ
σ_{bB90°	C377	17	90,9	8,2	9,04	+0,490	-0,791	highly certain
	C38C	25	86,5	11,9	13,77	-0,576	-1,121	highly certain
	C420	18	79,2	9,2	11,68	-1,166	+0,040	highly certain
	2T125B	11	98,1	14,7	14,97	-0,392	-0,692	chance
	2T131CR	71	109,0	12,7	11,73	-0,982	-0,727	highly certain
	2T131R	108	98,3	14,4	14,69	-0,146	-0,626	chance
	2T137D	114	104,3	16,5	15,84	-2,150	+1,213	certain
	2T146DR	24	106,9	10,2	9,53	-0,309	-0,467	chance
	2T147E	6	106,3	13,7	12,94	+0,435	-0,604	chance
	All measured values	394	100,7[x]	15,9	15,88	-1,545	-0,886	

[x] The average value from all samples equals average value μ of the entity.

Fiber type: 130SC/10 000

Resin type: Code 69

values could be checked.

The statistic data of the characteristic values determined for laminates from these three prepregs are compiled in Table 4. As the quotients of slope and curvature show, a normal distribution can be assumed for all random samples so that again it can be checked whether the average value of the random samples comes from a single basic entity. As basic entity we assumed the average value μ_1 of the results determined on samples with three prepregs and the average value μ_2 of all results.

As the next-to-last column shows, the deviation of average values $\bar{x}_i$ from μ_1 is arbitrary both for the longitudinal bending strength as well as for the /11 cross bending strength This means that prepreg preparation apparently provides no significant contribution to the total scattering. As the last column shows, this is no longer the case when the average value of random samples is compared with a basic entity which also considers measurements of samples with fibers and resin from other batches. From this we can conclude that the supposed systematic error differences in the results from Tables 2 and 3 are attributable primarily to the causes of "fibers" and "resin." This statement is supported by the results of Fig. 2, where the average values from random samples from the same resin and different fiber batches or the same fiber and different resin batches are illustrated. With one exception (Fig. b), we clearly see the influence of fiber and resin on the measured results.

Owing to the results presented above, both parameters must be considered as primary causes for the considerable amount of scattering of strength values for CFC.

Another possibly significant cause of scattering is the hardening of the prepreg gel to laminate. There are at least three reasons which make an optimum hardening doubtful:

a) difference in resin (state) for different resin batches;
b) different temperature distribution in the autoclave, above all, in large autoclaves;
c) deviations from a preset temperature, pressure, or vacuum profile for hardening, e.g., due to manual control.

TABLE 4. CHARACTERISTIC STATISTIC VALUES OF LONGITUDINAL BENDING AND CROSS BENDING STRENGTHS OF HM-FIBER LAMINATES FROM PREPREGS WITH EQUAL FIBER AND EQUAL RESIN

Mechanical properties	Prepreg batch	No. of samples	Average values $\bar{x}$ [N/mm²]	Stand.-deviation [N/mm²]	variation coefficient v [%]	quotient of slope	quotient of curvature	deviation $\bar{x}$ from μ_1	deviation $\bar{x}$ from μ_2
$\sigma_{bB0°}$ [N/mm²]	1885	24	1076,3	88,7	8,24	-0,203	-0,753	chance	certain
	1911	18	1136,0	107,7	9,48	+0,205	-0,366	chance	chance
	1921	23	1118,1	165,7	14,00	+0,562	-1,322	chance	chance
	All measured values from 2CM128B	101	1101,2	126,6	11,51	+2,05	-0,851	-	-
	All measured values from 200SC	250	1144,3	149,2	13,04	+1,773	-2,044	-	-
$\sigma_{bB90°}$ [N/mm²]	1885	41	85,4	10,5	12,32	-0,409	-0,584	chance	highly certain
	1911	24	80,2	11,1	13,83	+0,856	-0,459	chance	not certain
	1921	24	80,9	9,3	11,50	+0,561	+1,736	chance	certain
	All measured values from 2CM128B	88	83,6	10,3	12,42	+0,214	-0,331	-	-
	All measured values from 200SC	231	74,5	12,6	16,99	+0,8	-0,934	-	-

1) μ_1 = average value from all samples from one fiber batch

2) The average value from all samples equals μ_2 of the basic entity

Fiber type: 200SC/10 000

Resin type: Code 69

Resin batch No.: 83/8916

Fiber batch No.: 2CM128B

Differences in resin can influence the following important characteristic quantities for optimum hardening: /12

-- percent volatiles

-- gel point or gel range

-- viscosity behavior

-- resin flux

With regard to point b) above, in a large autoclave temperature differences up to 30°C should be expected during the heating phase. In a consideration of the available results, it was not possible, however, to prove the influence of parameters which make optimum hardening doubtful, because a study of point a) above was not performed and attention was not paid to the position of the laminate in the autoclave during the hardening.

As Fig. 3 shows, an influence of hardening is quite clear, however, since with the exception of run numbers 40 and 64 (leak in the vacuum), all hardening cycles were performed according to regulation. The laminates which were hardened in run numbers 41 and 43 were defective inasmuch as the individual prepreg gels were laminated by means of a teflon spatula. A decrease in those strengths determined by the resin or adhesion between fiber and resin is enormous and undeniable.

Since teflon is a separating agent, the laminates laminated with it are representative for fouling and improper handling in setting the individual layers.

From the discussion of the results presented above, one can conclude that the considerable scattering characteristic values of CFC are caused to a considerable extent by the following parameters:

-- fibers /14

-- resin

-- handling of the prepreg

-- hardening

The fraction of the total scattering due to the individual causes can only be estimated at the moment, for the reasons mentioned above. In consideration of additional experiences presented below, it can be assumed that fiber and hardening play a greater role in the total scattering than the other two.

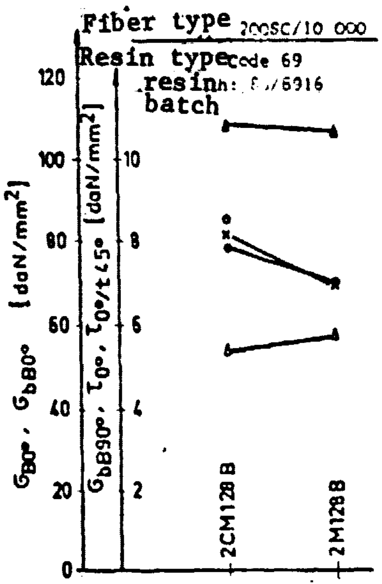

Fig. a

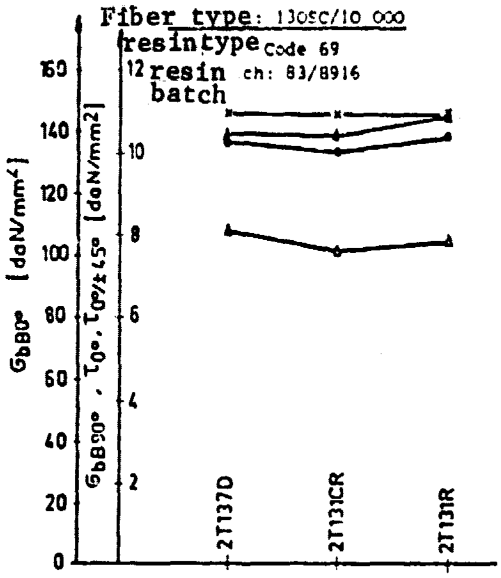

Fig. b

× = $\sigma_{bB90°}$

▲ = $\tau_{0°/\pm45°}$

▲ = $\sigma_{bB0°}$

o = $\sigma_{B0°}$

• = $\tau_{0°}$

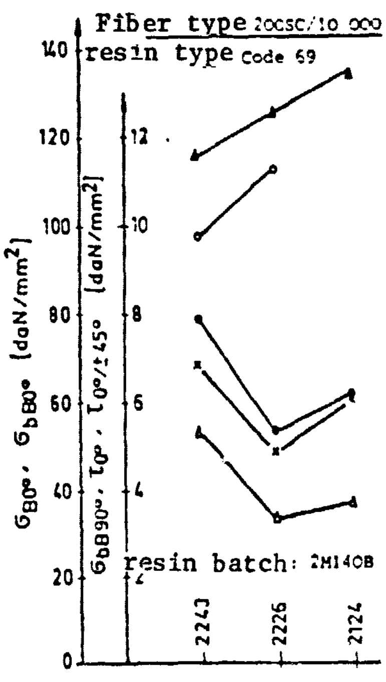

Fig. c

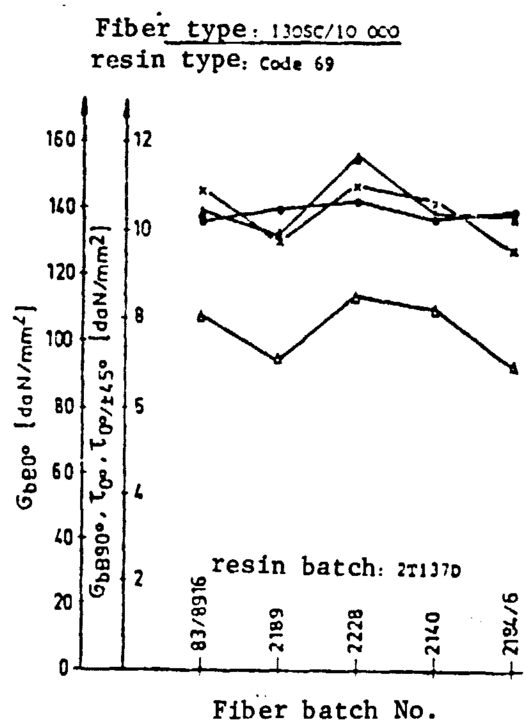

Fig. d

Fig. 2. Influence of fiber and resin batch on strength of CFC laminates.

As regards the problem of the fibers, it has come to light as a result of these studies that the degree of surface pretreatment was not held constant or could not be held constant.

The fibers are subjected to a preliminary surface treatment in order to improve adhesion of fibers with the resin. This preliminary surface treatment affects not only the adhesion between fiber and resin, as Fig. 4 shows, but also the tensile and bending strength. The percentage of total scattering due to the fibers will depend at least on the consistency of preliminary surface treatment.

Besides this parameter, there are certainly other effects, caused by the fiber processing, which affect fiber properties and thus contribute to this scattering. From Fig. 5 we can see, for instance, that the tensile strength of C-fiber filaments have become larger since 1975 on the average, and their scattering has become smaller. Scattering of the variation coefficients was also less in this period and the fraction of smaller coefficients was greater. Nevertheless, the extent of scattering is still considerable. If we assume, for instance, that the greatest variation coefficient can be correct both for the /18 smallest and greatest average value, then the limits of strength are about 230daN/mm^2 and 410daN/mm^2 with a statistical certainty of 99.7%. That this consideration is correct is demonstrated by the fact that Toray Co. gives a guaranteed minimum tensile strength value of 230 daN/mm^2 in their data sheet for the fiber T300.

Summary and Outlook for Possibilities to Reduce the Scattering of Characteristic CFC Values

Previously, in prepreg specifications, only a check of prepreg properties and of the hardened laminate was required. Obviously, this is no longer sufficient.

A check of the two components, fiber and resin, is also important. As regards the fibers, the fiber manufacturer must insure that the degree of preliminary surface treatment is kept within certain limits. This means that the degree of preliminary surface treatment must be controllable and specified.

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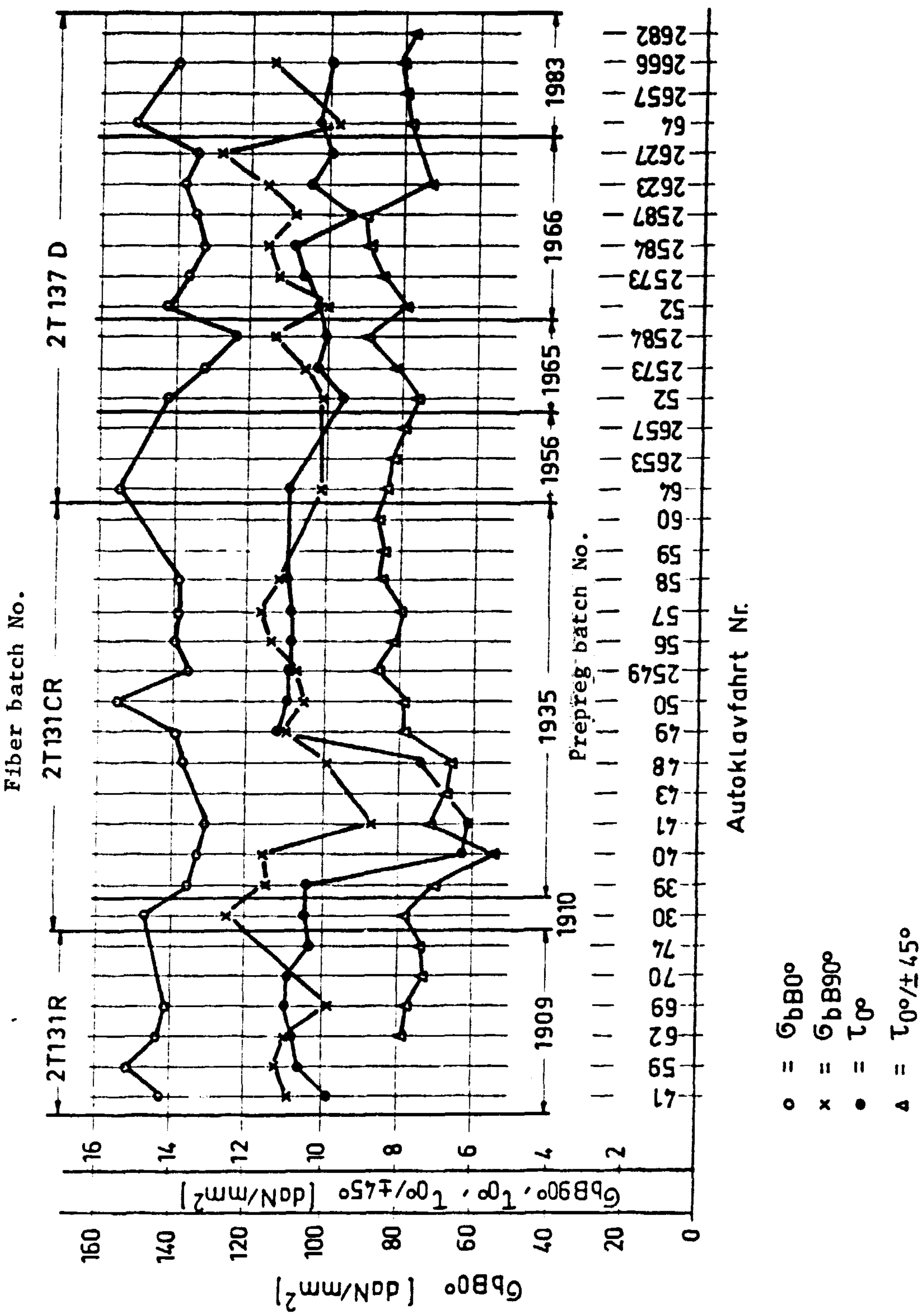
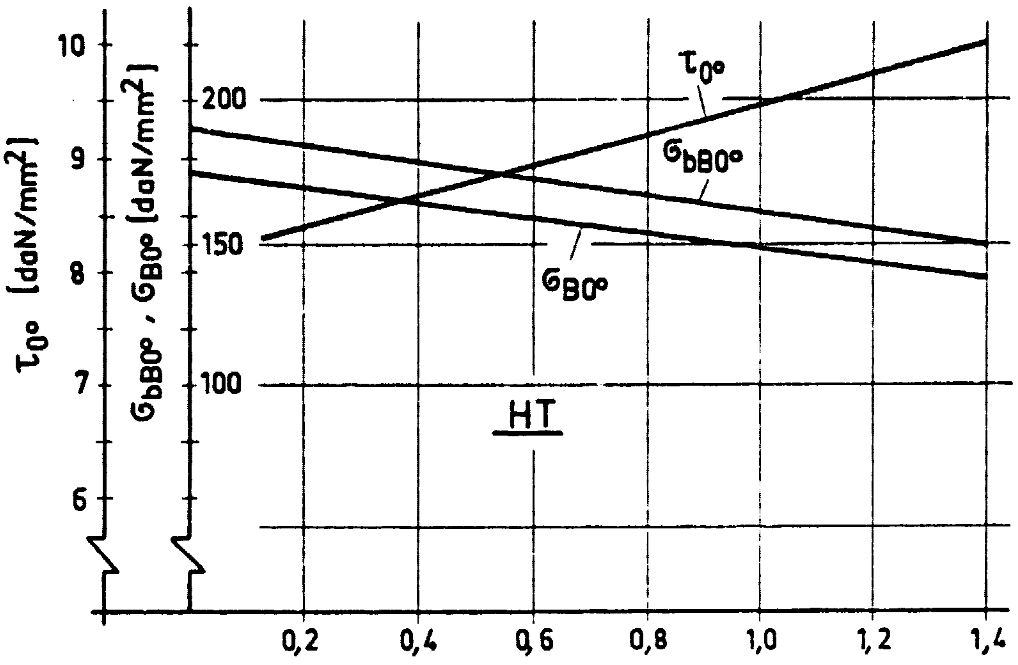
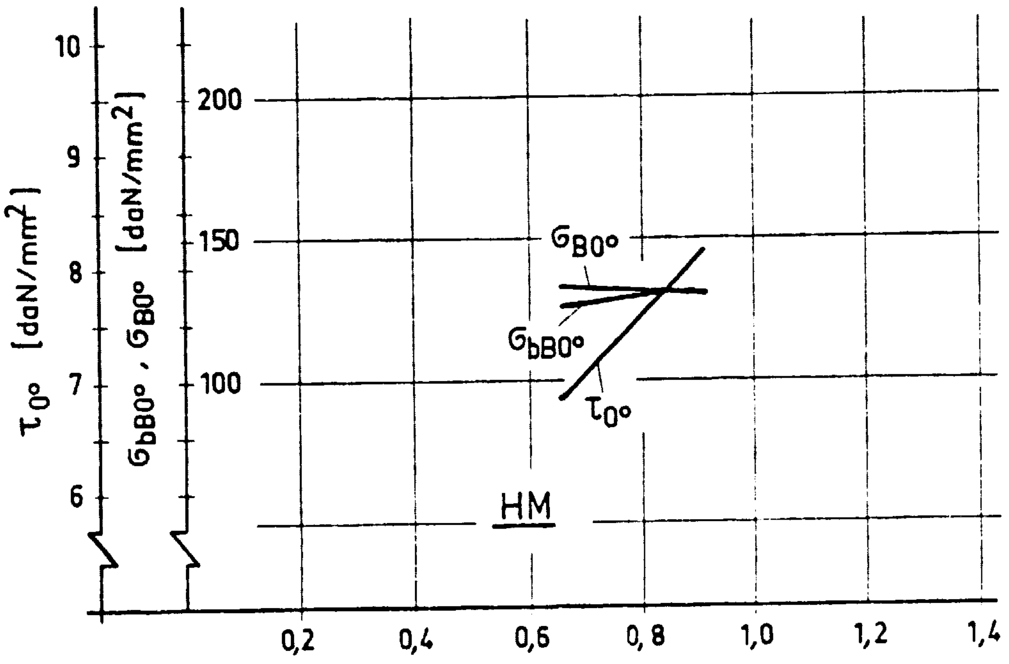


Fig. 3. Influence of autoclave runs on the strength of CFC laminates.
Fiber type: 130SC/10 000



Degree of preliminary fiber surface treatment



Degree of preliminary fiber surface treatment

Fig. 4. Strengths of CFC as a function of preliminary fiber surface treatment.

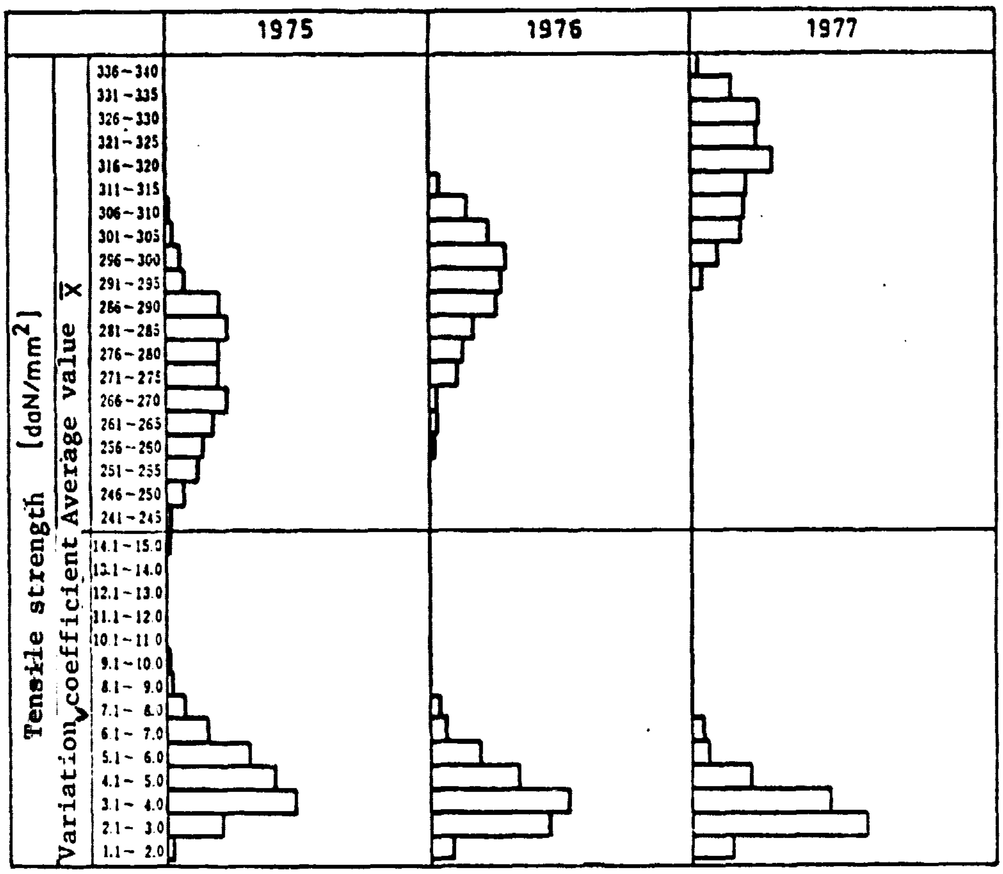


Fig. 5. Average values and variation coefficients of tensile strengths of C-fiber filaments (report from Toray Conference in Europe, 1978).

Since different preliminary surface treatments are used by fiber manufacturers and normally no details of these methods are made known, this problem can only be solved through cooperation between manufacturer and consumer.

Since, as has been shown, the properties of composites in and perpendicular to the fiber direction will depend on the degree of preliminary surface treatment, this treatment should be designed so that optimum conditions are present for the basic strengths $\sigma_{\parallel}$, $\sigma_{\perp}$ and $\tau_{\#}$ with regard to practical composites (0°/±45°/90°). This information has nothing to do with scattering -- it simply shows one possibility for improving the compatibility of fiber composites.

Another, relatively simple, possibility for reducing the scattering is a /19
specific selection of fibers of certain quality by fiber manufacturers. The certainly increased fiber costs would have to be compared to the effects of a reduced scattering.

In principle, for the resin components, the same information will apply as pertained to fibers, i.e., the state of the resin should be controllable and specified. In addition, it should be assured that hardening of the prepreg is optimum.

Since no methodical study has been performed up to now, but rather measured results occurring within a certain time period were used for the above discussion, no quantitative statement can be made.

The overall problem and consequences connected with it should be solved or quantified by a methodical study. Such a program could be designed as follows:

1. Study of resins and fibers from different prepreg charges;

2. Preparation of laminates with the different prepreg charges, each with the same hardening cycle;

3. Determination of the extent of faults (cavities, cracks) with non-destructive test methods;

4. Determination of characteristic mechanical values of the laminates from point 2 above, at room temperature, 150°C and after aging (warm, moist climate);

5. Determination of causes of failure of the test samples from point 4 above;

6. Checking the degree of hardening of the hardened laminate; /20

7. Creation of correlations between all experiences gained from points 1 to 5 above;

8. Preparation of laminates with the different prepreg charges, each with different hardening cycles;

9. Like points 3 to 6 above;

10. Creation of correlation between the experiences gained from point 9 above;

11. Working out suggestions to reduce the extent of scattering.